

formed, yielding 47% $P_4C_6B_2NH_2H_2O$, an anhydride of tetryl boric acid, m.p. 140-51° sol. in water only with heating. Addn. of 6 g. PhCl/CH₂ to EtMgBr from 7 g. EtBr followed by heating 2 hrs., addn. of 1.82 g. KBF₄, heating 1.5 hrs., cooling and quenching in satd. NH₄Cl gave after treatment of the ppt. and the org. layer with concd. NH₄OH, 27% of $P_4C_6B_2NH_2H_2O$ III, which can be crystd from H₂O with some hydrolysis. With an NH₄OH soln. of CuCl in Et₂O II gave 51% phenylacetylide in 98% yield. Treatment of III with LiAlD₄ gave 97% $P_4C_6B_2NH_2D_2$. Et₂O III, m.p. 100-105°, treatment with Et₂O/BF₃ Et₂O gave 90% Et₂O III, m.p. 84°. Et₂O III is unstable. The latter reacts with pyridine readily and in a few minutes at 0° yields up to 90% III.

"APPROVED FOR RELEASE: 03/14/2001

CIA-RDP86-00513R001447520003-5

Revised by telephone 10-14-86
by [unclear]
T-100P [unclear] Attn: [unclear] 10-14-86
[unclear] T-100P [unclear] Attn: [unclear] 10-14-86

APPROVED FOR RELEASE: 03/14/2001

CIA-RDP86-00513R001447520003-5"

In CCl_4 gave a brown ppt. contg. Ph_3B -pyridine salt and Cu salts; evapn. of the filtrate and heating with H_2O 2 hrs. with $\text{Ca}(\text{OH})_2$ and powd. Fe gave BzOH . The brown ppt. extd. with Me_2CO gave on extn. with Me_2CO and treatment of the insol. part with aq. KI , a ppt. of CuI and iodine; the filtrate gave triphenylboron pyridine salt, m. 212-18° (decompn.).

G. M. Kosolapoff

Distr: 4E3d/LE4j

Tetra(2-thienyl)boropotassium. V. A. Sazonova and
E. P. Serебряков. U.S.S.R. 106,396, Aug. 25, 1957.
The title compd. is obtained by the action of halo-2-thienyl-
magnesium on KBF_4 . An aq. soln. of the product is used
for detn. of Cs and Rb in a soln. of their salts. M. Hosh

2/2 Card A, V. A.

NDK

AUTHORS: Nesmeyanov, A. N., Sazonova, V. A.,
Drozd, V. N.

62-11-15/29

TITLE: On the Magnesium-Organic Compound of the Selenophene
(O magniyorganicheskom soyedinenii selenofena).

PERIODICAL: Izvestiya AN SSSR, Otdelenie Khimicheskikh Nauk, 1957,
Nr 11, pp. 1389-1391 (USSR)

ABSTRACT: In connection with the investigation of tetraarylboronic salts and the investigation of the reaction-process-possibility in the case of potassiumborofluoride with RMgX in the heterocyclic series here experiments were carried out in order to obtain iodide- α -selenenile-magnesium. It became evident that this metalorganic compound develops in an ester-solution from the α -iodoselenophene and magnesium when applying ethylene bromide as reaction accompanying matter. The iodide- α -selenenile-magnesium enters reaction with CO₂, benzophenone and produces α -selenophenecarbonic acid or diphenil- α -selenenilecarbinol respectively. In the reaction with potassiumborofluoride tetra-(α -selenenile)boropotassium

Card 1/2

SAZONOVA, V.A.; SEREBRYAKOV, E.P.; KOVALEVA, L.S.

Synthesis and analytical properties of tetra(α -thienyl)boric and tetra(p-anisyl)boric salts of alkali metals. Dokl. AN SSSR 113 no.6:1295-1298 Ap '57. (MLRA 10:6)

1. Moskovskiy gosudarstvennyy universitet im. M.V. Lomonosova.
Predstavлено академиком A.N. Nesmeyanovym.
(Alkali metal borates) (Anisile)

SOV/62-59-1-28/38

5(3)

AUTHORS:

Nesmeyanov, A. N., Sazonova, V. N., Drozd, V. N.

TITLE:

Organic-Boron Heterocyclic Compounds (Bororganicheskkiye geterotsiklicheskiye soyedineniya)

PERIODICAL:

Izvestiya Akademii nauk SSSR. Otdeleniye khimicheskikh nauk, 1959, Nr 1, pp 163 - 166 (USSR)

ABSTRACT:

In the present communication the authors report that the reaction RMgX with potassium fluoborate earlier investigated (Ref 2) was extended to oxygen-containing heterocyclic compounds (furan, sylvan) and triheterocycl boron compounds in the form of complexes were synthesized with pyridine. Tetra-(2-furyl) and tetra-5(2-methylfuryl)boron anions were obtained in the form of different salts. The presence of furan and sylvan nuclei in these anions was confirmed by decomposition of organo-boron compounds with alkali. Therein 2-chloro-mercuri furan and 2-methyl-5-chloro-mercuri furan were formed. Tetra-5-(2-methylfuryl)boron potassium separates ions of cesium, rubidium and quaternary ammonium salts. Pyridine salts of the type

Card 1/2

 $[C_5H_5^+NH]B^-Ar_4$ proved to be appropriate for the transition to

Organo-Boron Heterocyclic Compounds

SOV/62-59-1-28/38

tri(heterocyclic) boron compounds. The heating of pyridine salts in alcohol is sufficient for the synthesis of tri-substituted boron compounds in the form of pyridinates. In an exchange reaction between tetra-(2-selenyl)boron potassium and pyridine chlorine hydrate the pyridinate of triselenyl boron is immediately formed. There are 2 references, 1 of which is Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova
(Moscow State University imeni M. V. Lomonosov)

SUBMITTED: May 30, 1958

Card 2/2

5(2),5(3)

AUTHORS:

Sazonova, V. A., Leonov, V. N.

SOV/75-14-4-18/30

TITLE:

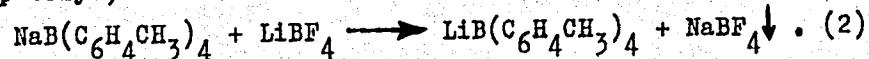
Tetra-(p-tolyl)-boron-lithium as a Reagent for the Quantitative Determination of Sodium

PERIODICAL: Zhurnal analiticheskoy khimii, 1959, Vol 14, Nr 4, pp 483-484
(USSR)

ABSTRACT: Utilizing a paper, published earlier (Ref 2), which dealt with the reaction of magnesium-organic compounds with sodium boron fluoride, the authors found that in the reaction of p-tolyl-magnesium bromide with sodium boron fluoride tetra-(p-tolyl)-sodium boride is formed which is difficultly soluble in water:



This compound reacts in an alcoholic solution with lithium boron fluoride and, based on one exchange-reaction, the water-soluble tetra-(p-tolyl)-boron lithium is formed:



$\text{LiB(C}_6\text{H}_4\text{CH}_3)_4$ forms colorless crystals, easily soluble in water, alcohol, acetone, and ether, insoluble in petroleum ether.

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Tetra-(p-tolyl)-boron-lithium as a Reagent for the
Quantitative Determination of Sodium

SOV/75-14-4-18/30

Tetra-(p-tolyl)-boron lithium precipitates in aqueous solution the cations Na^+ , K^+ , Rb^+ , Cs^+ and others. The authors used for the quantitative determination of sodium a 10-15% aqueous solution of this reagent adding 2 - 3 drops of a 3% NaCl -solution. On the addition of NaCl solution a precipitate is formed by which the reagent is saturated with tetra-(p-tolyl)-boron sodium. The solution is subsequently filtered and is then ready for use. In the determinations of sodium the authors added to the 1 ml NaCl solution, which also contained 0.0301 g of NaCl per ml, 3 - 4 ml of the reagent solution. The formed precipitate was sucked off after 5 - 10 minutes, it was washed 4 - 5 times with 1.5 - 2 ml of the reagent solution and dried in an exsiccator at room temperature over P_2O_5 . The precipitate was then freed from the remaining reagent by 4 - 5-time washing with absolute ether and dried once more at room temperature until weight constancy was attained. A table gives the results obtained in 4 determinations made by this method. The reagent also precipitates K^+ , Rb^+ , and Cs^+ which can be quantitatively precipitated with tetraphenyl boron lithium already before the determination of sodium, the sodium remaining in solution. A great advantage of the method

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Tetra-(p-tolyl)-boron-lithium as a Reagent for the
Quantitative Determination of Sodium

SOV/75-14-4-18/30

described is that only small amounts of the reagent are needed, because it can be re-transformed into $\text{LiB}(\text{C}_6\text{H}_4\text{CH}_3)_4$ according to equation (2) after the gravimetric determination of the tetra-(p-tolyl)-boron sodium. The method additionally permits the determination of sodium in the presence of lithium. Aqueous solutions of tetra-(p-tolyl)-boron lithium cannot be kept for a long time in the air without change. Therefore, the reagent has to be prepared immediately before using it. The two steps (equations (1) and (2)) for the preparation of the reagent are described in every detail. There are 1 table and 3 references, 2 of which are Soviet.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova
(Moscow State University, imeni M. V. Lomonosov)

SUBMITTED: June 24, 1958

Card 3/3

5 (2,3)

AUTHORS: Nesmeyanov, A. N., Academician,
Sazonova, V. A., Drozd, V. N.

SOV/20-126-5-25/69

TITLE: Ferrocenyl Boric Acid and 1,1' Ferrocenylene-Diboric Acid and
Their Reactions (Ferrotsenilbornaya i 1,1'-ferrotsenilendi-
bornaya kisloty i ikh reaktsii)

PERIODICAL: Doklady Akademii nauk SSSR, 1959, Vol 126, Nr 5, pp 1004 - 1006
(USSR)

ABSTRACT: The authors have obtained a mixture of the two acids mentioned
in the title by the action of a mixture of lithium and dilithium-ferrocene (Ref 1) on boron-n-butyl-ester. They were extracted by alkalies out of the reaction mixture. Their separation is explained by the high degree of solubility of the mono-acid and the insolubility of the diboric acid in ether. With respect to its chemical properties the mono-acid is similar to arylboric acid. Thus, ferrocene is obtained by hydrolysis in the presence of $ZnCl_2$; with sublimate ferrocene-mercury chloride is easily formed (Ref 1), whilst with cupric chloride and cupric bromide chlorine-ferrocene and bromine ferrocene are formed (Ref 2). In the same manner also the dihalogen-ferrocene

Card 1/2

Ferrocenyl Boric Acid and 1,1' Ferrocenylene-Diboric Acid and Their Reactions

SOV/20-126-5-25/69

derivatives are produced from the diboric acid mentioned in the title. Thus, all three mono-halogen-ferrocenes: chlorine-, bromine-, and iodine-ferrocene are known, as well as all three hetero-annular dihalogen ferrocenes. The present investigation has revealed the properties of the dibromo ferrocene which was obtained in a purer crystalline form under the action of the ferrocenylene diboric acid. Under the interaction of ferrocenyl-boric acid and of an ammoniacal silver oxide solution, ferrocene and diferrocnyl are produced, whilst for the phenyl boric acid a hydrolysis up to benzene under the action of this reagent and for the alkyl-boric acids a doubling of the radical and a disproportioning of the latter had been known. There are 2 Soviet references.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova
(Moscow State University imeni M. V. Lomonosov)

SUBMITTED: April 16, 1959

Card 2/2

5.3700(B)

67317
SOV/20-129-5-27/645(7)
AUTHORS: Nesmeyanov, A. N., Academician, Sazonova, V. A., Drozd, V. N.TITLE: OxyferrocenePERIODICAL: Doklady Akademii nauk SSSR, 1959, Vol 129, Nr 5,
pp 1060 - 1063 (USSR)ABSTRACT: No oxy-derivative of ferrocene has been hitherto known. The authors obtained oxyferrocene in two ways, namely, by way of ferrocenyl acetate: 1) by mixing aqueous solutions of ferrocenyl boric acid (Ref 3) with copper acetate, ferrocenyl acetate (yield 59%) and diferrocenyl (21%) are obtained, ferrocenyl propionate and diferrocenyl are formed with copper propionate (see Scheme); 2) ferrocenyl acetate was formed on heating bromo-ferrocene with copper acetate. The ferrocenyl acetate structure was confirmed by the reaction with phenyl magnesium bromide (see Scheme). The methyl diphenyl carbinol sample obtained in this connection melts with pure methyl-diphenyl-carbinol without a reduction in the melting point. Ferrocenyl benzoate was separated from the oxyferrocene yield after benzoylation. Oxy-ferrocene (ferrocenol) is easily separated from alkaline solutions, ✓
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Oxyferrocene

SOV/20-129-5-27/64

much like phenol, on bubbling CO₂ through them. Oxyferrocene is a yellow crystalline air-unstable substance. It can be recrystallized from water (with quick heating), but becomes somewhat darker. It is soluble in ether, alcohols and chloroform. Moreover, the authors obtained the following derivatives of oxyferrocene: ferrocenyl benzoate, ferrocenyl ester of benzene sulfonic acid as well as oxyferrocene methyl ether (Table 1). All ethers and esters are crystalline substances which readily solve in organic solvents. Further properties of oxy-ferrocene are being investigated. There are 1 table and 5 references, 2 of which are Soviet.

4

SUBMITTED: September 11, 1959

Card 2/2

53700(B)

6861

5(3)

AUTHORS:

Nesmeyanov, A. N., Academician,
Sazonova, V. A., Drozd, V. N.S/020/60/130/05/021/061
B011/B005

TITLE:

Some Reactions of the Halogen Derivatives of Ferrocene.
Ferrocenylamine. Ferrocenylacetate

PERIODICAL:

Doklady Akademii nauk SSSR, 1960, Vol 130, Nr 5, pp 1030-1032
(USSR)

ABSTRACT:

The purpose of the paper is the synthesis of ferrocenylamine, ferrocenylacetate, N-ferrocenylphthalimide, N-acetylferrocenylamine, and ferrocene carboxylic acid nitrile. The paper continues previous investigations by the authors (Ref 1, together with E. G. Perevalova and O. A. Nesmeyanova: Refs 2,3). As further investigations have shown, the exchange of the halogen in halogen ferrocenes by reaction with the salt of the bivalent (and monovalent $Cu_2(CN)_2$) copper is a method generally applicable for substitution of the halogen atom in the ferrocene ring by copper-bound radicals. The substitution of the halogen in halogen ferrocenes by an acetoxy group proceeds even more smoothly than previously described by the authors in aqueous-

Card 1/3

68611

Some Reactions of the Halogen Derivatives of
Ferrocene. Ferrocenylamine. Ferrocenylacetate

S/020/60/130/05/021/061
B011/B005

alcoholic solutions by boiling with copper acetate for 15 min. The yield in ferrocenylacetate is 90% (see Scheme). N-ferrocenylphthalimide is formed by heating (135-140°) the mixture of halogen ferrocene with copper phthalimide (see Scheme). By the action of hydrazine hydrate, the N-ferrocenylphthalimide is easily transformed into ferrocenylamine with a total yield of 50%. The ferrocenylamine was identified as N-acetylferrocenylamine. The halogen may also be replaced by the phthalimide group by boiling in an aqueous-alcoholic solution for 2 h. The method described here for preparing ferrocenylamine is certainly more convenient than the methods described in references 4 and 5. Bromoferrrocene reacts with copper cyanide, and gives a good yield in ferrocene carboxylic acid nitrile. The reaction proceeds less readily with chloroferrrocene. Previously, the nitrile was prepared by other methods (Refs 6,7). There are 8 references, 5 of which are Soviet.

ASSOCIATION:
Card 2/3

Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova
(Moscow State University imeni M. V. Lomonosov)

Some Reactions of the Halogen Derivatives of
Ferrocene. Ferrocenylamine. Ferrocenylacetate

68611
S/020/60/130/05/021/061
B011/B005

SUBMITTED: November 12, 1959

Card 3/3

57700(B)

80000
S/020/60/131/05/029/069
B011/B117AUTHORS: Nesmeyanov, A. N., Academician,
Sazonova, V. A., Drozd, V. N., Nikonova,
L. A.TITLE: 1-(1'-Halogenferrocenyl) Boric Acids in the Synthesis of Ferrocene Derivatives

PERIODICAL: Doklady Akademii nauk SSSR, 1960, Vol 131, Nr 5, pp 1088-1091 (USSR)

TEXT: The authors proved in their paper that the reaction of 1,1'-ferrocenylene diboric acid with cupric chloride or bromide performed in a mixture of benzene and water yields 1-(1'-chloroferrrocenyl) and 1-(1'-bromoferrocenyl) boric acid. Cupric chloride or cupric bromide must, however, be used in a quantity corresponding to one $B(OH)_2$ group. The structures of 1-(1'-halogenferrocenyl) boric acids were established by means of the preparation of the corresponding halogenferrocenes by hydrolysis in the presence of zinc salts. The 1-(1'-halogenferrocenyl) boric acids react in a similar way to the aryl boric acids with mercury salts, yielding the corresponding mercury compounds of ferrocene: 1-(1'-chloroferrrocenyl) mercury chloride and 1-(1'-bromoferrocenyl) mercuric bromide. They are easily symmetrized by sodium thiosulfate to yield di-1-(1'-chloroferrrocenyl) mercury and di-1,1-(1'-bromoferrocenyl) mercury. From the two last-mentioned substances, the authors prepared 1'-chloro-1-iodoferrrocene and

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80000

1-(1'-Halogenferrocenyl) Boric Acids in the
Synthesis of Ferrocene Derivatives

S/020/60/131/05/029/069
B011/B117

1'-bromo-1-iodoferrocene which have hitherto been unknown. The procedure used was the same as the one described for ferrocenyl mercury chloride (Ref 2). When an attempt was made to prepare heterocyclic chlorobromoferrocene by reacting cupric chloride with 1-(1'-bromoferrocenyl) boric acid, 1,1'-dichloroferrrocene (cf Scheme) was obtained. The latter reaction confirms the simple substitution of the halogen in the ferrocene ring in the presence of copper salts, which has previously been established by the authors (Ref 3). There are 3 Soviet references.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova
(Moscow State University imeni M. V. Lomonosov)

SUBMITTED: January 7, 1960

Card 2/2

81724

S/020/60/133/01/35/070
B011/B003

53700(B)

AUTHORS: Nesmeyanov, A. N., Academician, Sazonova, V. A.,
Drozd, V. N., Nikonova, L. A.

TITLE: Oxyferrocenes and Their Derivatives

PERIODICAL: Doklady Akademii nauk SSSR, 1960, Vol. 133, No. 1,
pp. 126 - 129

TEXT: Ferrocenyl-allyl ester is easily formed by heating oxyferrocene with allyl bromide in acetone in the presence of potash. By heating ferrocenyl-allyl ester at 215-220°C in nitrogen, it is decomposed up to oxyferrocene. Part of the ester remains unchanged. Claisen regrouping could not be carried out with ferrocenyl-allyl ester (Ref. 2). The authors' considerations on the instability of the "quinoid" state in the ferrocene molecule were confirmed by experiments on the oxidation of 1,1'-dioxyferrocene with air. The molecule decomposes and separates an inorganic iron compound. The resulting cyclopentadienone was isolated as a dimer. Furthermore, the authors compared the dissociation constants of oxyferrocene and phenol, and described 1,1'-dioxyferrocene

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S/020/60/133/01/35/070
B011/B003

Oxyferrocenes and Their Derivatives

and its derivatives. The pH-values of 0.005 M solutions of oxyferrocene or phenol in 5% alcohol, which had partly been neutralized with NaOH up to 30, 50, and 70%, were measured at 17°C by means of a glass electrode and an JJT -5 (LP-5) potentiometer. Table 1 lists the values obtained for oxyferrocene. It shows that oxyferrocene is a weaker acid than phenol. The authors synthesized 1,1'-dioxyferrocene derivatives by using 1,1'-ferrocenylene boric acid. This acid reacts with copper acetate and forms 1,1'-ferrocenylene diacetate in a 41% yield. 1,1'-dioxyferrocene ester is obtained in a yield of 83% if a $\text{B}(\text{OH})_2$ group has previously been substituted by a halogen in this acid. When copper acetate acts upon 1-(1'-ferrocenyl halide) boric acids (synthesis: Ref. 5), the acetoxy group substitutes both the halogen and the $\text{B}(\text{OH})_2$ group. 1,1'-dibromoferrocene may also be used for the synthesis of ferrocenylene diacetate (cf. Scheme: X denotes the halogen). The frequencies characteristic of the unsubstituted ferrocene ring are missing in the infrared spectrum of ferrocenylene diacetate. Hydrolysis of the first-mentioned compound (in a nitrogen atmosphere) and subsequent acidification or blowing through of CO_2 yields yellow needles of 1,1'-dioxyferrocene. *44*

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Oxyferrocenes and Their Derivatives

S/020/60/133/01/35/070
B011/B003

which is highly sensitive to air (cf. Scheme). The alkaline hydrolyzate could be used for synthesizing 1,1'-dioxyferrocene derivatives, i.e., 1,1'-dimethoxyferrocene, 1,1'-ferrocenylene dibenzoate, 1,1'-ferrocenylene dibenzosulfonate, and O,O'-(1,1'-ferrocenylene) diglycolic acid. All these derivatives are stable in air. There are 2 tables and 7 references: 2 Soviet, 4 American, and 1 Swiss.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova
(Moscow State University imeni M. V. Lomonosov)

SUBMITTED: April 8, 1960

4

Card 3/3

NESMEYANOV, A.N.; SAZONOVA, V.A.; DROZD, V.N.

Reactions of halo derivatives of ethyl- and acetylferrocene.
Dokl. AN SSSR 137 no.1:102-105 Mr-Ap '61. (MIRA 13:12)

1. Moskovskiy gosudarstvennyy universitet im. M.V.Lomonosova.
(Ferrocene)

33263
S/062/62/000/001/002/015
B106/B101

53700

AUTHORS: Nesmeyanov, A. N., Sazonova, V. A., and Drozd, V. N.

TITLE: Influence of the carboxyl and carbomethoxy groups on the substitution of halogen in ferrocene compounds

PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeniye khimicheskikh nauk, no. 1, 1962, 45 - 47

TEXT: This work has been undertaken to ascertain whether halogen in ferrocene derivatives is easily substituted in the presence of carboxyl and carbomethoxy groups. Oxidation of 1'-bromo-1-acetylferrocene with iodine in absolute pyridine yielded 1'-bromoferrocene-1-carboxylic acid (recrystallized from aqueous alcohol, m. 154-158°C (decomposition), 41% yield). The bromium of this compound is completely exchanged for the acetoxy group when boiling with copper acetate in aqueous alcohol for 15 min. The yield of 1'-acetoxyferrocene-1-carboxylic acid (yellow crystals from benzene-hexane, m. 126-130°C (decomposition), readily soluble in water) was 60% of the theoretical value. The reaction product did not contain initial carboxylic acid. (The nephrite test was negative before

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33263

S/062/62/000/001/002/015

B106/B101

Influence of the carboxyl and ...

purification of the product). The methyl ester of 1'-bromoferrocene-1-carboxylic acid (recrystallized from n-hexane, m. 68-69.5°C), obtained by esterification of the acid with diazomethane in 96% yield, reacts less readily with copper acetate under the same conditions than the relevant acid. After 30-min reaction, chromatography on aluminium oxide revealed 45% of the initial methyl ester besides the expected product, the methyl ester of 1'-acetoxyferrocene-1-carboxylic acid (m. 45.5-47°C, 16% yield). The methyl ester of 1'-acetoxyferrocene-1-carboxylic acid is partially decomposed during the chromatographic process. The fact that the carboxyl group, contrary to other electron-acceptor groups, increases the mobility of a halogen bound to the ferrocene system is explained as follows: The copper salt of 1'-bromoferrocene-1-carboxylic acid, formed at the beginning of the reaction, forms a coordinate bond, Cu...Br, whereby the C-Br bond is polarized and the halogen may be exchanged for the acetoxy group. This behavior is similar to the aromatic carboxylic acids ortho-substituted by halogen, which, in the presence of copper compounds, also exchange the halogen readily for nucleophilic groups. These facts can not be attributed to simple activating effects of the carboxyl group since the halogen is not as reactive in the relevant benzene carboxylic acids para-substituted by

X

Card 2/3

S/020/62/147/003/025/027
B101/B186

AUTHORS: Nesmeyanov, A. N., Academician, Sazonova, V. A.,
Gerasimenko, A. V.

TITLE: α -pyridyl-ferrocene and 1,1'-di-(α -pyridyl)-ferrocene

PERIODICAL: Akademiya nauk SSSR. Doklady, v. 147, no. 3, 1962, 634 - 635

TEXT: The following syntheses of pyridine compounds of ferrocene, and reactions of these compounds, are described: (1) Ferrocene and a small amount (0.03 g from 3 g initial substance) of α -pyridyl ferrocene, and 87 - 89°C, were obtained by heating 1,1'-ferrocenylene-diboric acid with ether and chromatographic separation on aluminum oxide. (2) 24% α -pyridyl ferrocene was obtained by reaction of ferrocenyl-lithium and 1,1'-dilithium ferrocene mixtures dissolved in ether, under an N_2 atmosphere, followed by extraction with ether carbonate in pyridine under an N_2 atmosphere, followed by extraction with ether and chromatographic separation on aluminum oxide. (3) Oxidation of α -pyridyl ferrocene and potassium permanganate in an acid medium was found to yield picolinic acid. (4) A red deposit of α -pyridyl ferrocene hydro-

Card

Card 1/2

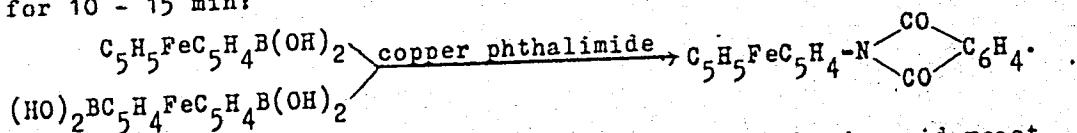
s/062/62/000/011/014/021
B101/B144

AUTHORS: Nesmeyanov, A. N., Sazonova, V. A., Gerasimenko, A. V., and
Medvedeva, V. G.

TITLE: Reactions of ferrocenyl boric acids with copper phthalimide

PERIODICAL: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh
nauk, no. 11, 1962, 2073 - 2074

TEXT: The following reactions were carried out with ferrocenyl boric acid (I) and 1,1'-diferrocenylene diboric acid (II) in boiling pyridine for 10 - 15 min:



Styryl boric acid, phenyl boric acid, and p-anisyl boric acid react similarly with copper phthalimide. Compounds obtained: N-ferrocenyl phthalimide, yield from I: 47 %, from II: 29 %, m.p. 156 - 156.5°C; N-styryl phthalimide, yield 56 %; N-phenyl phthalimide, yield 19 %; and

Card 1/2

Reactions of ferrocenyl boric...

S/062/62/000/011/014/021
B101/B144

N-p-methoxy phenyl phthalimide, yield 34 %.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova
(Moscow State University imeni M. V. Lomonosov)

SUBMITTED: June 4, 1962

Card 2/2

REF ID: A6572

NESEMEYANOV, A.N.; SAZONOV, V.A.; GERASIMENKO, A.V.; MEDVEDEVA, V.G.

Reaction of ferroceneboronic acids with copper phthalimide.
Izv. AN SSSR. Otd.khim.nauk no.11:2073-2074 N '62. (MIRA 15:12)

1. Moskovskiy gosudarstvenny universitet im. M.V. Lomonosova.
(Ferroceneboronic acid) (Phthalimide)

Sazonova, V. A.

AID Nr. 982-1 4 June

DIFERROCENYLS AND TERFERROCENYLS (USSR)

Nesmeyanov, A. N., V. N. Drozd, V. A. Sazonova, V. I. Romanenko, A. K. Prokof'yev, and L. A. Nikonova. IN: Akademiya nauk SSSR. Izvestiya. Otdeleniye khimicheskikh nauk, no. 4, Apr 1963, 667-674.

S/062/63/000/004/012/022

A series of substituted diferrrocenyls, 1,1'-diferrrocenylferrocene, also named 1,1'-terferrrocenyl (I), and higher homologues were synthesized at the Moscow State University imeni M. V. Lomonosov by the reaction of a mixture of

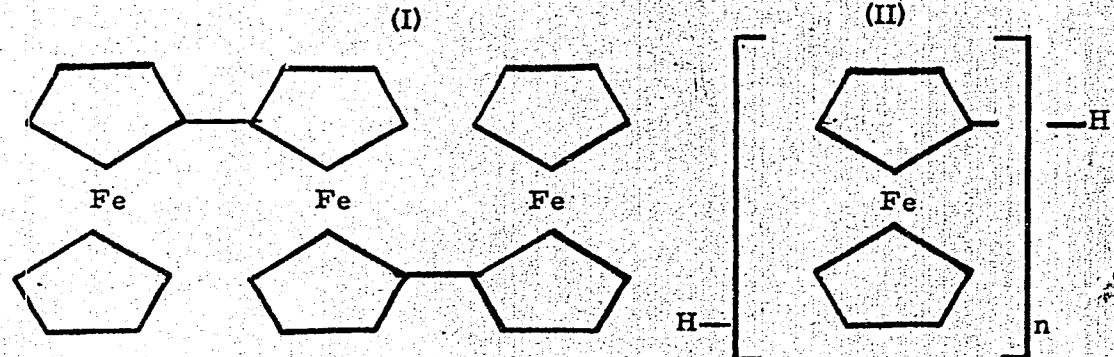
Card 1/4

AID Nr. 982-1 4 June

DIFERROCENYLS AND TERFERROCENYLS [Cont'd]

S/062/63/000/004/012/022

bromoferrocene and 1,1'-dibromoferrocene with copper at 105-120°C. The following products were isolated by Al_2O_3 chromatography: ferrocene, di-ferrocenyl, 1,1'-terferrocenyl with the structure I and homologues II, in which $n < 4$:



The 1,1'-polyferrocenylenes obtained were diamagnetic. The derivatives of difericenyl and terferrocenyl were also obtained by application of the general

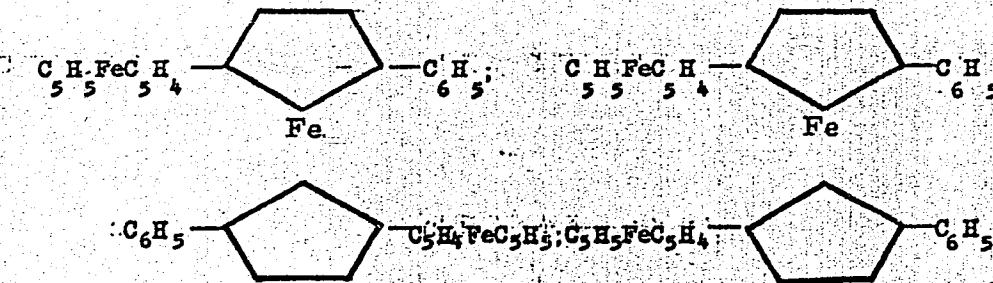
Card 2/4

AID Nr. 982-1 4 June

DIFERROCENYLS AND TERFERROCENYLS [Cont'd]

S/062/63/000/004/012/022

method for synthesizing ferrocenes, that is, by using substituted cyclopentadienes (in this case, ferrocenylcyclopentadienes) as the starting materials. The synthesis of 3-ferrocenyl-1-phenylcyclopentadiene (III) was achieved by the condensation of acetylferrocene with the ethyl β -benzoylpropionate in the presence of sodium ethylate; III yielded a substituted terferrocenyl - 1,1'-diferrocenyl-3,3'-diphenyl-ferrocene (IV) - after being treated first with sodium amide in liquid ammonia and then with ferrous chloride. Anti and syn structures are ascribed to IV, which could also be in the racemic and meso forms:



Card 3/4

AID Nr. 982-1 4 June

DIFERROCENYLS AND TERFERROCENYLS (Cont.)

S/062/63/000/004/012/022

Investigation of IR spectra indicated that bands with frequencies of 1000 and 1113 cm^{-1} are characteristic for the system of cyclopentadiene rings bound together in disubstituted diferrocenyls which contain no free cyclopentadiene rings.

[BN]

Card 4/4

RESMEYANOV, A.N., akademik; SAZONOVA, V.A.; GERASIMENKO, A.V.

α -Pyridylferrocene and 1,1'-di-(α -Pyridyl)-ferrocene. Dokl.
AN SSSR 147 no. 3:634-635 N '62. (MIRA 15:12)

1. Moskovskiy gosudarstvennyy universitet im. M.V. Lomonosova.
(Ferrocene) (Pyridine)

SAZONOVA, V.A.; KARPOV, V.I.

Tetra(1-indolyl) boron salts. Zhur.ob.khim. 33 no.10:3313-
3315 O '63. (MIRA 16:11)

L 10088-63

EWP(j)/EPF(c)/EWT(m)/EDS

Pc-4/Pr-4 EM/WW/MAY

ACCESSION NR: AP3000302 -

S/0020/63/150/001/0102/0104

AUTHOR: Nesmeyanov, A. N. (Academician); Drozd, V. N.; Sazonova, V. A.TITLE: Diazo ferrocene compounds64
63

SOURCE: AN SSSR. Doklady*, v. 150, no. 1, 1963, 102-104

TOPIC TAGS: diazo, ferrocene, acidolysis, diazoamino, diazcaminoferrocene, ferrocenediazonium, [(phenyldiazonamino)cyclopentadienyl]cyclopentadienyliron, 1, 1'-ferrocenylenebisdiazonium

TEXT: Diazo derivatives of ferrocene have been prepared by acidolysis of diazoamino derivatives and their properties studied. Treatment of diazoaminoferrocene with concentrated HCl at -40 to -20C produced a violet solution which gave off nitrogen at temperatures as low as -15C. The presence of (chlorocyclopentadienyl)- and (aminocyclopentadienyl)-cyclopentadienyliron in the solution indicated the formation of a ferrocenediazonium cation intermediate. The existence of this cation was

Card 1/b2

L 10088-63

ACCESSION NR: AF3000302

O
confirmed by the fact that similar treatment of [(phenyldiazoamino)-cyclopentadienyl]cyclopentadienyliron (I) also produced a violet solution of ferrocenediazonium. The last reacts with 2-naphthol to form the dark-green dye 1-ferrocenazo-2-naphthol. The fact that 1-phenylazo-2-naphthol was found among the acidolysis products of I shows that the tautomeric equilibrium of I is shifted toward 1-ferrocenyl-3-phenyltriazene. Ferrocenediazonium can undergo nucleophilic substitution in an HX solution ($X = Cl, Br, I$): nitrogen evolution began at temperatures as low as -15°C and stopped at -5°C, and the (halocyclopentadienyl)cyclopentadienyliron was formed in a yield greater than 70%. Treatment of bis[1-(phenyldiazoamino)cyclopentadienyl]iron with concentrated HCl at -40 to -20°C gave a dark-violet solution containing 1, 1'-ferrocenylenebis diazonium. Orig. art. has: 5 formulas and 1 table.

ASSOCIATION: Moscow State University

Card 2/ β_2

L 12919-63 EPF(c)/EWP(j)/EWT(m)/BDS ASD Pr-4/Pc-4 RM/WW/MAY
ACCESSION NR: AP3000519 S/0020/63/150/002/0321/0324

AUTHOR: Nesmeyanov, A. N.; Drozd, V. N.; Sazonova, V. A.

65
64

TITLE: Azides of ferrocene

SOURCE: AN SSSR Doklady, v. 150, no. 2, 1963, 321-324

TOPIC TAGS: ferrocene, azide, triazocyclopentadienyliron, (bromocyclopentadienyl)cyclopentadienyliron, bis(1-bromocyclopentadienyl)iron, (aminocyclopentadienyl)cyclopentadienyliron, bis(1-aminocyclopentadienyl)iron, triazole, triazene, diazonium salt, bis(1-phenyldiazoaminocyclopentadienyl)iron

ABSTRACT: (Triazocyclopentadienyl)cyclopentadienyliron (I) was obtained in a nearly quantitative yield by reaction of N_3^- ions with (bromocyclopentadienyl)cyclopentadienyliron for 48 hours in aqueous N, N -dimethylformamide at room temperature. Boiling bis(1-bromocyclopentadienyl)iron in aqueous alcohol for six minutes produced bis(1-triazocyclopentadienyl)iron (II) in a 31% yield. Both reactions were conducted in the presence of Cu^{2+} ions. Compounds I and II are yellow crystalline substances melting at 53-54 and 58.5-59°C, respectively, readily soluble in organic solvents, and decomposing on heating and in light. The low yield of II is explainable by the thermal instability of this compound.

Card 1/2

L 12919-63

ACCESSION NR: AP3000519

The use of the known tolylsulfonyl azide method gave lower yields of I and II. The reduction of I with aluminum lithium hydride produced a 72% yield of (aminocyclopentadienyl)cyclopentadienyliron, previously obtained for the first time by two other methods at the authors' laboratory. A similar reduction of II produced bis(1-aminocyclopentadienyl)iron, which is unstable and oxidizes rapidly in air. In regard to the chemistry of the ferrocene azides, the addition of I to a strained double bond, such as that in dimethyl exo-cis-3, 6-endo-oxy- Δ_4 -tetrahydropthalate, to form the corresponding triazoline and the formation of triazenes of the ferrocene series are mentioned. Such ferrocene triazenes as bis(1-phenyldiazoaminocyclopentadienyl)iron were used for preparing diazonium salts of the ferrocene series by acidolysis. Orig. art. has: 8 formulas and 7 tables.

ASSOCIATION: Moskovskiy gosudarstvenny universitet im. M. V. Lomonosova
(Moscow State University)

SUBMITTED: 23Jan63

DATE ACQ: 12Jun63

ENCL: 00

SUB CODE: CH

NO REF Sov: 003

OTHER: 006

Card 2/2

NESMEYANOV, A.N., akademik; SAZONOV, V.A.; ROMANENKO, V.I.

Alkylation of ferrocenylamine. Dokl. AN SSSR 152 no. 6:1358-
1359 O '63. (MIRA 16:11)

1. Moskovskiy gosudarstvennyy universitet im. M.V. Lomonosova.

L 27266-65 EWT(m)/EPF(c)/EMP(j) pc-4/pr-4 RM

ACCESSION NR: AP4010758

S/0020/64/154/001/0158/0159

AUTHORS: Nesmeyanov, A.N. (Academician); Sazonova, V.A.; Drozd,
V.N.

TITLE: Substitution of halide in bromoferrocene by aromatic and heterocyclic radicals

SOURCE: AN SSSR. Doklady*, v. 154, no. 1, 1964, 158-159

TOPIC TAGS: halide, bromoferrocene, nitrogen, phenylferrocene, p-tolylferrocene, α -thienylferrocene, N-pyrrylferrocene, N-indolylferrocene, ferrocenylphenylacetylene, copper derivatives

ABSTRACT: By heating bromoferrocene with a mixture of pyrrol-sodium and Cu_2Br_2 , N-pyrrylferrocene is obtained in a yield of 17%. The absence of absorption bands at $3400-3440\text{ cm}^{-1}$ in the spectra of N-indolylferrocene and N-pyrrylferrocene, characteristic for the NH bond of the pyrrol ring, confirms their structure as N-substituted compounds. Copper phenylacetylene reacts with bromoferrocene in boiling methyl formamide to form ferrocenyl phenyl acetylene. The

Card 1/2

L 27266-65

ACCESSION NR: AP4010758

experimental process for obtaining phenyl ferrocene,⁷ p-toly
ferrocene, α -thienyl ferrocene, N-pyrryl ferrocene and N-indyl ferro-
cene and ferrocenyl phenyl acetylene from bromoferrocene under ni-
trogen is described. Orig. art. has: 4 equations and 1 table.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova
(Moscow State University)

SUBMITTED: 20Jul63

ENCL: 00

OTHER: 001

Card 2/2

ACCESSION NR: AP4019977

S/0020/64/154/006/1393/1394

AUTHORS: Nesmeyanov, A.N. (Academician); Sazonova, V.A.; Drozd, V.N.

TITLE: Decomposition of alpha-ferrocenylcarbonic ions to fulvenes

SOURCE: AN SSSR. Doklady*, v. 154, no. 6, 1964, 1393-1394

TOPIC TAGS:decomposition, alpha ferrocenylcarbonic ion, fulvene, diphenyl ferrocenylcarbinol, ferrocene derivative, diphenylfulvene

ABSTRACT: Since N-methyl-2-ferrocenylpyridine hydroxide decomposes in sunlight to form N-methyl-2-cyclopentadienylide-pyridine, cyclopentadiene and Fe^{2+} , it is hypothesized that the positive charge in the atom, combined with a ferrocene molecule, weakens the iron carbide bond. This hypothesis is shown to be true for certain α -ferrocenylcarbonic ions. The increased facility of their formation from corresponding carbinols and their esters is known because of the stabilizing effect of the free electronic vapor of iron. The carbide cations, being formed from diphenylferrocenylcarbinol and 1,1-bis(-hydroxy-benzhydryl)-ferrocene in acetic acid in the presence of HCl, are exceptionally unstable and decomposed in

1/2
Card

NESMEYANOV, V.N., akademik; SAZONOVA, V.A.; ROMANENKO, V.I.; RODIONOVA,
N.A.; ZOL'NIKOVA, G.P.

Photolysis of ferrocene derivatives. Dokl. AN SSSR 155 no. 5:
1130-1133 Ap '64. (MIRA 17:5)

1. Moskovskiy gosudarstvenny universitet im. M.V.Lomonosova.

L-11864-65

EWT(m)/EPF(c)/EPR/EWP(j) Pcl-1/Pr-1/Ps-1 RPL RM/WW

ACCESSION NR: AP4043548

S/0020/64/157/004/0922/0925

AUTHOR: Nesmeyanov, A. N.; Sazonova, V. A.; Romanenko, V. I.

TITLE: Derivatives of ferrocenylamine

SOURCE: AN SSSR. Doklady*, v. 157, no. 6, 1964, 922-925

TOPIC TAGS: ferrocenylamine, ferrocenylamine derivative, synthesis, diphenylferrocenylamine, tertiary aromatic amine, monophenylferrocenylamine, secondary aromatic amine, azoferrocene, oxidation, chloroferrocnylamine, dichloroazoferrocene, chloroferrocnylamine purification

ABSTRACT: Several derivatives of ferrocenylamine were synthesized and their properties were studied. Bromoferrocene reacted with sodium diphenylamine in the presence of CuBr to form diphenylferrocenylamine, a typical tertiary aromatic amine. Bromoferrocene reacted with sodium acetanilide in the presence of CuBr to form monophenylferrocenylamine (I). The secondary amine I was not extracted by dilute HCl, but formed the chlorohydrate with concentrated

Card 1/2

L 11864-65

ACCESSION NR: AP4043548

HCl and the benzoyl derivative with benzoyl chloride. Ferrocenylamine (II) reacted with iodobenzene in the presence of CuI to form azoferrrocene (III), and not I. II was oxidized quantitatively to III by atmospheric oxygen in benzene solution at room temperature in the presence of CuBr or CuCl. Substituents on the ferrocene nucleus effected its rate of oxidation. Thus 1-(1'-chloroferrocenyl) amine (IV) oxidized to dichloroazoferrocene more slowly than II oxidized to III. IV is a yellow crystalline material, melting at 116C; its acetyl derivative melts at 124.5C. It can be obtained from 1-(1'-chloroferrocenyl)boric acid and copper phthal imide. Since IV oxidized much more slowly, it can be readily purified of II. Orig. art. has: 5 equations.

ASSOCIATION: None

SUBMITTED: 29Apr64

ENCL: 00

SUB CODE: OG, MT

NO REF SOV: 004

OTHER: 002

Card 2/2

L 24829-65 EWT(m)/EPF(c)/EPR/EWP(j) - Pc-Li/Pr-Li/Ps-Li RPL RM/uW

ACCESSION NR: AP4049920

S/0020/64/159/003/0591/0594

27

24

β

AUTHOR: Drozd, V. N.; Sazonova, V. A.; Nesmeyanov, A. N. (Academician)

TITLE: Ferrocenyl sulfones. Ferrocenyl mesityl sulfone under the conditions of the Smiles rearrangement

SOURCE: AN SSSR. Doklady*, v. 159, no. 3, 1964, 591-594

TOPIC TAGS: sulfone, ferrocenyl sulfone, Smiles rearrangement, quinoid state, butyllithium, ferrocene, mesityl sulfone

ABSTRACT: It is known that substitution by the S_N2 mechanism in the aromatic series presupposes an intermediate quinoid state. The authors assumed that reactions of this type could not occur in the case of ferrocene, and used the recently discovered Smiles rearrangement of o-methyl diaryl sulfones in the presence of n-butyl-lithium to test this hypothesis. Since the Smiles rearrangement proceeds via an intramolecular S_N2 mechanism, it was expected that ferrocenyl mesityl sulfone would not undergo a rearrangement. To prepare sulfones of the ferrocene series, the authors reacted bromoferrrocene with copper salts of aryl sulfinic acids:

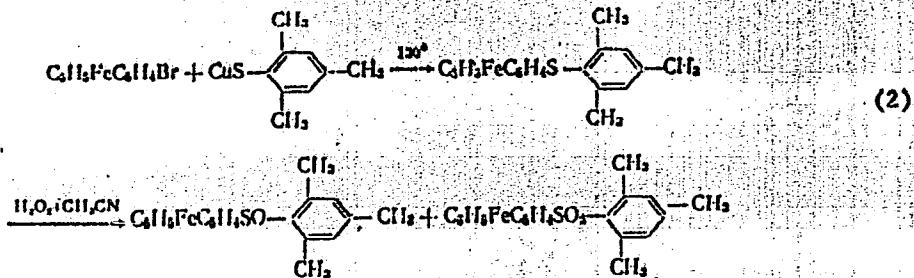


Card 1/3

L 21829-65

ACCESSION NR: AP4049920

To prepare ferrocenyl mesityl sulfone, however, another method of synthesis had to be found:



It was found that when ferrocenyl mesityl sulfide was treated with n-butyllithium, a side methyl group of the sulfone was substituted by lithium. No products of the Smiles rearrangement were found in the experiments. The data therefore cast a doubt on the possibility of a nucleophilic S_N2 substitution in the ferrocene series. The procedures used in the synthesis of phenyl ferrocenyl sulfone, diferrocenyl sulfone, ferrocenyl mesityl sulfone, and ferrocenyl mesityl sulfide,

Card 2/3

L 24829-65

ACCESSION NR: AP4049920

in the oxidation of the latter, and in the reaction of ferrocenylmesityl sulfone with n-butyllithium are described in detail. "T. Yu. Frid helped with the experimental work." (Orig. art. has: 3 chemical equations.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova
(Moscow State University)

SUBMITTED: 02Jul64

ENCL: 00

SUB CODE: CC

NO REF Sov: 002

OTHER: 008

Card

3/3

136639-65 EWT(m)/EPF(c)/EWP(j)/EMA(c) Pe-4/Pr-4 RM
ACCESSION NR: AP5001993 S/0020/64/159/006/1334/1337 3/2

AUTHOR: Nesmeyanov, A. N. (Academician); Drozd, V. N.; Sazonova, V. A.; Postnov, V. N.

TITLE: Certain properties of ferrocene diazo compounds

SOURCE: AN SSSR. Doklady, v. 159, no. 6, 1964, 1334-1337

TOPIC TAGS: ferrocene diazo compound; ferrocenyldiazonium cation; reactivity

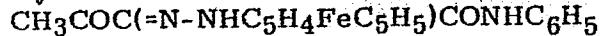
ABSTRACT: The chemical properties and reactivity of the ferrocenyldiazonium cation (A), obtained by acidolysis of benenediazoaminoferrrocene (B), were investigated. It was determined that A was less active than the phenyldiazonium cation in nitrogen coupling reactions due to the greater nucleophilicity of the ferrocene in comparison to benzene. The acidolysis of B is a reversible reaction: treatment of the reaction mixture with pyridine gave the initial triazene. A reacted with diethylamine to form 3,3-diethyl-1-ferrocenyltriazine; with a mixture of methyl-aniline and pyridine it formed 3-methyl-3-phenyl-1-ferrocenyltriazine; and with β -naphthylamine, A formed β -naphthalenediazoaminoferrrocene,

Card 1/2

L-36639-65

ACCESSION NR: AP5001993

β -C₁₀H₇-N=N -NH₂C₆H₄FeC₅H₅. The latter did not give A upon treatment with concentrated HCl. B reacted fairly rapidly with β -naphthol at 100C to form 1-ferroceneazo-2-naphthol, and with acetylacetone to form 3-ferrocenylhydrazone-acetylacetone. B reacted more slowly with acetoacetic ester to form the α -ferrocenyl-hydrazoneacetoacetic ester and its anilide



Reaction of B with acetoacetic acid anilide, however, gave the β -anilanilide CH₃C(=NC₆H₅)C(=NNHC₆H₄FeC₅H₅)CONHC₆H₅. An aqueous solution of the potassium ferrocenyldiazotate was obtained by slowly mixing ferrocenyldiazonium chloride and KOH at -30 to -20C and then heating slowly. Orig. art. has: 3 equations

ASSOCIATION: Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova
(Moscow State University)

SUBMITTED: 08Jun64

ENCL: 00

SUB CODE: OC, GC

NR REF SOV: 002

OTHER: 000

Card 2 / 2

NESMEYANOV, A.N., akademik; SAZONOVA, V.A.; DROZD, V.N.

Introduction of aromatic and heterocyclic radicals to ferrocene.
Reaction of bromoferrocene with organomagnesium compounds. Dokl.
AN SSSR 163 no.3 575-577 N '65. (MIRA 18:II)

L. Moskovskiy gosudarstvenny universitet im. M.V. Lomonosova.

L 27405-65 EWT(m)/EPF(c)/EPR/EWP(j) Pe-L/Pr-L/Ps-L RPL RM/WW

ACCESSION NR: AP5004596 S/0020/65/160/002/0355/0358

AUTHOR: Nesmeyanov, A. N. (Academician); Sazonova, V. A.; Drozd, V. N.
Rodionova, N. A.

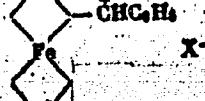
4
25
P

TITLE: Alpha-ferrocenylcarbonium salts

SOURCE: AN SSSR. Doklady, v. 160, no. 2, 1965, 355-358

TOPIC TAGS: ferrocene, ferrocenyl carbonium salt, ferrocenyl phenylcarbinol, electron spin resonance, biphenyl, tetraphenylborate

ABSTRACT: The authors synthesized two relatively stable ferrocenylphenylcarbonium salts - the perchlorate and tetraphenylborate - from ferrocenylphenylcarbinol in an acetic acid solution by the action of HClO_4 and tetraphenylborosodium, respectively:



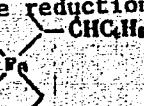
, where x is ClO_4^- or $\text{B}(\text{C}_6\text{H}_5)_4^+$. and studied some of their properties. The authors found from electron spin resonance data that ferrocenylphenylcarbonium perchlorate and diphenylferrocenylcarbonium perchlorate are diamagnetic, which is in agreement with the carbonium-ion.

Card 1/2

L 27405-65

ACCESSION NR: AP5004596

structure. It is possible that the reduction of the ferrocenylphenylmethyl cation to the radical



(which immediately undergoes pairing) takes place merely by heating with the tetra-phenylborate anion (biphenyl was produced). The preparation of all the compounds synthesized in the experiment is described. Orig. art. has: 5 chemical formulas.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova (Moscow state university)

SUBMITTED: 02Aug64

ENCL: 00

SUB CODE: OC

NO REF SOV: 001

OTHER: 009

Card 2/2

I. 53998-65

EWT(m)/EPF(c)/EWP(j)/EWA(c)

Pc-4/Pr-4

RPL JW/RM

UR/0020/65/161/005/1085/1088

ACCESSION NR: AP5011531

AUTHORS: Nesmeyanov, A. N. (Academician); Sazonova, V. A.; Romanenko, V. I.TITLE: Diferrocenylamine

SOURCE: AN SSSR. Doklady, v. 161, no. 5, 1965, 1085-1088

TOPIC TAGS: amine group, alkylation, degradation reaction, acetylene, reduction, ferrocene

ABSTRACT: The authors have previously reported on obtaining n-ethyl-ferrocenylamine and n-phenylferrocenylamine by heating the sodium derivative of n-acetyl-ferrocenylamine with bromoferrrocene in the presence of Cu_2Br_2 . All experimental attempts to hydrolyze amide to differrocenylamine have been unsuccessful. This differs from results with n-acetylphenylferrocenylamine. When boiled in alcohol with KOH, n-acetylferrocenylamine remains unchanged. When heated for a long time, it gradually decomposes. Acid hydrolysis leads rapidly to complete destruction of the ferrocene system. Diferrocenylamine may be obtained by reduction splitting of n-acetylferrocenylamine by lithium aluminum hydride. Splitting is dominant only when 0.25 mole of $LiAlH_4$ per mole of amide is used.

Card 1/2

L 53998-65

ACCESSION NR: AP5011531

With large excess of LiAlH₄, splitting and reduction produce comparable yields.

Diferrocenylamine resembles diphenylamine in some properties, as it is not extracted from benzene solutions by dilute acid, but is dissolved in concentrated HCl. It forms chlorohydrate when HCl is introduced into an ether solution of amine. Diferrocenylamine is alkylized by boron fluoride triethyl oxonium. During reduction of n-acetylferrocenylamine and n-benzoylferrocenylamine by lithium aluminum hydride, ethylferrocenylamine and benzylferrocenylamine respectively are formed, with corresponding yields of 94 and 86%. The alkylation of these by boron fluoride triethyl oxonium gives ethylbenzylferrocenylamine. Orig. art. has: 4 formulas.

ASSOCIATION: Moskovskiy gosudarstvennyy universitet im. M. V. Lomonosova
(Moscow State University)

SUBMITTED: 09Dec64

ENCL: 00

SUB CODE: OC, GC

NO REF SOV: 002

OTHER: 001

Card 2/2

1831
NESMEYANOV, A.N.; SAZONOV, V.A.; ROMANENKO, V.I.; ZOL'NIKOVA, G.P.

Photolysis of 1,1'-ferrocenedicarboxylic acid. Izv. AN SSSR.
Ser. khim. no.9:1694-1695 '65. (MIRA 18:9)

1. Moskovskiy gosudarstvennyy universitet.

NESMEYANOV, A.N.; DROZD, V.N.; SAZONOV, V.A.

Acetylation of N-acylaminoferroenes. Izv. AN SSSR. Ser. khim. no.7:
1205-1208 '65. (MIRA 18:7)

1. Moskovskiy gosudarstvennyy universitet im. M.V.Lomonosova.

L 16984-66 EWT(m)/EWP(j)/T WW/JW/JWD/RM

ACC NR: AP6002101

SOURCE CODE: UR/0062/65/000/011/2061/2063

AUTHORS: Nesmeyanov, A. N.; Sazonova, V. A.; Drozd, V. N.; Rodionova, N. A.; Zudkova, G. I.

ORG: Moscow State University im. M. V. Lomonosov (Moskovskiy gosudarstvennyy universitet)

TITLE: Properties of α -ferrocenylcarbonic ions

SOURCE: AN SSSR. Izvestiya. Seriya khimicheskaya, no. 11, 1965, 2061-2063

TOPIC TAGS: ferrocene, organic synthetic process, nuclear magnetic resonance

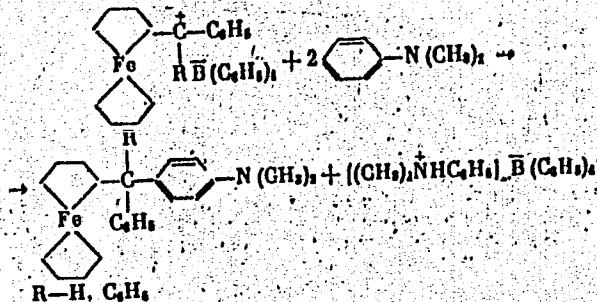
ABSTRACT: Reaction of tetraphenylborates of phenyl-(I) and diphenylferrocenyl-carbonates (II) with dimethylaniline (III) was investigated. Preparation of I and II and some of their properties were described by the authors in a previous work (Dokl. AN SSSR, 160, No. 2, 1965). The reaction described here takes place at 5-20°C within a few minutes and proceeds according to the equation

Card 1/2

UDC: 542.91+547.1'3+546.72

L 16984-66

ACC NR: AP6002101



The structures of the products were confirmed by NMR spectra. Preparation of p-dimethylaminophenylferrocenyl-, p-dimethylaminodiphenylferrocenyl-, and p-dimethylaminophenyldiferrocenylcarbinols is described. The authors express their gratitude to V. I. Sheychenko for working on the NMR spectra. Orig. art. has: 1 equation.

SUB CODE: 07 SUBM DATE: 24Mar65/ ORIG REF: 001 OTH REF: 001

Card 2/2 M95

L 27092-66

EWT(m)/EWP(j)

RM

SOURCE CODE: UR/0062/65/000/007/1205/1208

ACC NR: AP6017400

AUTHOR: Nesmeyanov, A. N.; Drodz, V. N.; Sazonova, V. A.

ORG: Moscow State University im. M. V. Lomonosov (Moskovskiy gosudarstvennyy universitet)

TITLE: Acetylation of N-acylaminoferroenes

SOURCE: AN SSSR. Izvestiya. Seriya khimicheskaya, no. 7, 1965, 1205-1208

TOPIC TAGS: organoiron compound, organic imine compound, amine, ferrocene, acetic anhydride

ABSTRACT: In studying acetylation in the ring of N-ferrocenylphthalimide and N-acetylferrocenylamine, the authors found that the acylamino-group is an electron acceptor with respect to ferrocene and directs substitution mainly in the free cyclopentadienyl ring. Thus, in the acetylation of N-ferrocenylphthalimide with acetic anhydride in the presence of H_3PO_4 , the main reaction product was 1'-(N-phthalimido)-1-acetylferrocene and a smaller amount of homoannular N-phthalimidoacetylferrocene (yields of 40 and 7% of theoretical, respectively). Acetylation of N-acetylferrocenylamine proceeds with the formation of large amounts of tarry substances; of the reaction products with 17% yield only 1'-acetamino-1'acetylferrocene was isolated. It was found that 1'-amino-1-acetylferrocene is obtained by the hydrolysis of 1'-(N-phthalimido)-1-acetylferrocene and 1'-acetaminoacetyl-ferrocene and from the Curzius reaction, from 1'-acetylferrocene-1-carboxylic acid. Orig. art. has: "1 formula." /JPRS/

SUB CODE: 07 / SUBM DATE: 10Jun63 / ORIG REF: 004 / OTH REF: 004
Card 1/1 UDC: 542.91+592.957+546.72

L 26575-66 EWT(m)/EWP(j)/T IJP(c) RM

ACC NR: AP6016974

SOURCE CODE: UR/0020/65/165/003/0575/0577

AUTHOR: Nesmeyanov, A. N. (Academician); Sazonova, V. A.; Drozd, V. N.ORG: Moscow State University im. M. V. Lomonosov (Moskovskiy gosudarstvennyy universitet)TITLE: Introduction of aromatic and heterocyclic radicals into ferrocene.⁴⁷ Reaction of bromoferrrocene with organomagnesium compounds⁴⁷

SOURCE: AN SSSR. Doklady, v. 165, no. 3, 1965, 575-577

TOPIC TAGS: ferrocene, organomagnesium compound, brominated organic compound, organic synthetic process, Grignard reagent, nuclear magnetic resonance, chromatography

ABSTRACT: In previous studies the authors synthesized hydroxyferrocene, 1,1'-dihydroxyferrocene, ferrocenylamine, diferrocenylamine, phenylferrocenylamine, diphenylferrocenylamine, ferrocenylazide (followed by conversion to diazo compounds of ferrocene), ferrocenylarylsulfones and other ferrocene derivatives through halo-derivatives of ferrocene. In this work the reaction of bromo-ferrocene with organomagnesium compounds was investigated. Phenyl-, alpha-thienyl-, and alpha-naphthylferrocenes were produced in 75-85% yield by the addition of an ether solution of Grignard reagent to a mixture of bromoferrrocene, copper bromide, and copper, followed by distillation of the ether and heating of the reaction mixture under nitrogen at 130°. In the case of an alicyclic Grignard reagent, cyclohexyl magnesium chloride, the reduction of bromoferrrocene to ferrocene became the basic reaction, and only cyclohexene was isolated from

Card 1/2

UDC: 547.257.2+547.254.6

L 26575-66

ACC NR: AP6016974

the reaction mixture. Pyrryl magnesium bromide reacted with bromoferrrocene in the presence of Cu_2Br_2 / Cu to form a mixture of alpha- and beta-pyrryl-ferrocenes, which were separated chromatographically on alumina by elution with a mixture of heptane-benzene (4:1); alpha-pyrrylferrocene is eluted first. The reaction of indolyl magnesium bromide resulted in the formation of alpha- and beta-indolylferrocenes. The structures of the compounds obtained were demonstrated by nuclear magnetic resonance studies. The authors thank V. I. Sheychenko, of the Laboratory of Physicochemical Research, Institute of Chemistry of Natural Compounds, AN SSSR, where the measurements of the nuclear magnetic resonance spectra were carried out. [JPRS] 2

SUB CODE: 07, 20 / SUBM DATE: , 28 May 65 / ORIG. REF: 002 / OTH REF: 004

Card 2/2

L 31361-66 EWP(j)/EWT(m) IJP(c) RM
ACC NR: AP6021101

SOURCE CODE: UR/0062/66/000/002/0357/0357

50

B

AUTHOR: Nesmeyanov, A. N.; Romanenko, V. I.; Sazonova, V. A.

ORG: Moscow State University im. M. V. Lomonosov (Moskovskiy gosudarstvennyy universitet)

TITLE: Basicity constants of amines of ferrocene

SOURCE: AN SSSR. Izvestiya. Seriya khimicheskaya, no. 2, 1966, 357

TOPIC TAGS: ferrocene, amine, solution acidity, potentiometer, titrimetry,

distillation, buffer solution, chemical neutralization, alkyl radical/pH - Mc

potentiometer

ABSTRACT: Basicity constants of ferrocenylamine, ethyl ferrocenylamine, diethylferrocenylamine, 1,1'-chloroferrocenylamine, and alpha-pyridylferrocene were determined by potentiometric titration in 80% (by weight) ethanol in 0.025 N HCl on a pH - Mc type potentiometer, with G 200B glass electrode. Solvents were prepared as follows: distilled water was twice redistilled over alkaline potassium permanganate in equipment protected with ascarite-filled tubes; purchased absolute alcohol was treated with sodium (6 grams of sodium per liter of alcohol) and distilled, and the first and last quarter portions were rejected. The titration method is conventional; placement of electrodes was in a biphthalate buffer (pH 4.01), temperature $22 \pm 1^\circ$, and amine concentration 0.005 M. Basicity constants of the amines were determined for three degrees of neutralization (35, 50 and 65%); for each point, 6-9 measurements were made; the spread of pH values did not exceed 0.05. Comparison of basicity constants of N-alkylated ferrocene amines with those of aromatic and aliphatic amines shows that in the ferrocene series the effect of the alkyl group is the same as in the aliphatic series.

Orig. art. has: 1 table. [JPRS]

SUB CODE: 07 / SUBM DATE: 02Jul65 / ORIG REF: 004 / OTH REF: 002

UDC: 541.124.7 + 547.233 + 546.72

Card 1/1 C

ACC NR: AP7012421

SOURCE CODE: UR/0062/66/000/011 2017/2019

AUTHOR: Nesmeyanov, A. N.; Sazonova, V. A.; Zudkova, G. I. Isayeva, L. S.

ORG: Moscow State University im. M. V. Lomonosov (Moskovskiy gosudarstvenny universitet)

TITLE: Alpha-ferrocenylcarbonium salts

SOURCE: AN SSSR. Izvestiya. Seriya khimicheskaya, no. 11, 1966, 2017-2019

TOPIC TAGS: hydrolysis, dimethylamine, acetic acid, inorganic salt

SUB CODE: 07

ABSTRACT: The influence of the dimethylamino group, situated in the p-position of the benzene ring bonded to a carbonium carbon upon the stability and reactivity of alpha-ferrocenylphenylcarbonium salts was investigated. Three salts were synthesized from the corresponding carbinols and tetraphenylborosodium in glacial acetic acid. Such salts were more stable than the carbonium salts not containing the dimethylamino group. Hydrolysis of phenylferrocenyl- and diphenylferrocenylcarbonium tetraphenylborates is instantaneous, whereas the corresponding tetraphenylborates containing the dimethylamino group are recovered unchanged. Other reactions of the salts synthesized were studied: alkylation of dimethylaniline in the p-position; reactions with piperidine,

Card 1/2

UDC: 542.91+547.1'3+542.957+546.72
092 1358

ACC NR: AP7012421

forming p-dimethylaminophenylferrocenylmethyl- and p-dimethylaminodiphenyl-
ferrocenylmethyl- substituted piperidines. Orig. art. has: 1 formula.
[JPRS: 40,422]

2/2

L 19493-65 EPF(c)/EPR/EWP(j)/EWT(m) Po-4/Pr-4/Pb-4 RPL RM/WV

ACCESSION NR: AP5002075

S/0079/63/033/006/2042/2045 1.

B

AUTHOR: Sazonova, V. A.; Gerasimenko, A. V.; Shiller, N. A.

TITLE: Phenylation of alpha,beta-unsaturated ketones with triphenylboron and phenylboric acid

SOURCE: Zhurnal obshchey khimii, v. 33, no. 6, 1963, 2042-2045

TOPIC TAGS: triphenylboron, phenylboric acid, phenylation, unsaturated ketone, copper acetoacetate, arylation

ABSTRACT: The authors phenylated α -benzalacetacetate, benzalbenzoylacetate and benzalbenzoylacetone by boiling them with triphenylboron or phenylboric acid in the presence of copper acetoacetate, diluting with water, and then extracting the saturated products (α -benzhydrylacetacetate, benzhydrylbenzoylacetate and α -benzhydryl- α -acetylacetone) with ether. In the case of triphenylboron, the yields were over 100%, indicating that more than one phenyl group from triphenylboron takes part in the reaction. Phenylation of these α,β -unsaturated ketones was in the 4-position, indicating a radical mechanism. In the case of m-nitrophenylboric acid, only m,m'-dinitrodiphenyl was obtained under these conditions. When benzalacetophenone and dibenzalacetone were used as starting compounds, it was difficult to isolate any products due to condensation reactions between the ketones

Card 1/2

L 19493-65
ACCESSION NR: AP5002075

and copper acetoacetate. "The phenylation of benzalacetoacetate was carried out together with V. G. Shubin, a student." Orig. art. has: 2 chemical formulas.

ASSOCIATION: none

SUBMITTED: 09May62

ENCL: 00

SUB CODE: 00

NO REF SOV: 001

OTHER: 006

Card 2/2

"APPROVED FOR RELEASE: 03/14/2001

CIA-RDP86-00513R001447520003-5

ROMANYUK, F.I.; PETROV, G.S. [deceased]; GOLUHEVA, A.N.; KARTASHEV, N.A.;
SAZONOV, V.M.; KAMENSKIY, I.V.; OGNEVA, N.Ye.

New methods for preventing the flow of reservoir waters into
wells being exploited. Trudy VNII no.16:106-127 '58.
(MIRA 11:12)

(Oil field flooding)

APPROVED FOR RELEASE: 03/14/2001

CIA-RDP86-00513R001447520003-5"

SAZONOVA, V. V.

Sazonova, V. V. and Afanas'yev, G. A. "On the problem of the disposition prophylactics of tuberculosis in children of pre-school age by the method of allergometry," In the index: 2nd author - Sazonova, V. A. Trudy Medinstituta (Izhev. gos. med. in-t), Vol. VII, 1949, p 283-89

SO: U-3950, 16 June 53, (Letopis, 'Zhurnal 'nykh Statey, No. 5, 1949).

DEMIDOV, P.; CHMYR', A.; SAZONOVA, Ye.

Effect of gamma rays on the technological properties of wheat
grain. Muk.-elev. prom. 28 no.12:3-4 D '62. (MIRA 16:1)

1. Odesskiy tekhnologicheskiy institut im. Lomonosova.
(Wheat) (Gamma rays—Industrial applications)

SAZONOVA, Ye.A.

~~Changes in the respiration of the whole organism and different organs induced by photodynamic substances at various stages of the individual development. Uch.zap. KHGU 53:189-194 '54.~~
(MIRA 11:11)

1. Kafedra fiziologii cheloveka i zhivotnykh Khar'kovskogo gosudarstvennogo universiteta imeni A.M. Gor'kogo.
(RESPIRATION) (AGE) (PHOTODYNAMIC ACTION)

SAZONOV A. Ye.

Changes with age in the level of oxidation processes in different organs. Uch.zap. KHGU 53:195-198 '54. (MIRA 11:11)

1. Kafedra fiziologii cheloveka i zhivotnykh Khar'kovskogo gosudarstvennogo universiteta imeni A.M. Gor'kogo.
(AGE) (OXIDATION, PHYSIOLOGICAL)

SAZONOVA, Ye.A.

Materials on the ontogenetic physiology of the Chinese tussah moth
(*Antheraea pernyi* G.-M.). Report No.2: Changes in the chemical
composition and oxidative processes. Uch.zap.KHGU 68:137-151
'56 (MIRA 11:11)

1. Kafedra fiziologii cheloveka i zhivotnykh Nauchno-issledovatel'skogo
instituta i biologicheskogo fakul'teta Khar'kovskogo ordena trudovogo
krasnogo znameni gosudarstvennogo universiteta imeni A.M. Gor'kogo.
(SILKWORMS--CHEMICAL COMPOSITION) (INSECTS--DEVELOPMENT)
(OXIDATION, PHYSIOLOGICAL)

SAZONOVA, Ye. A., Cand Biol Sci -- (diss) "Nature of the growth variations in oxidative processes in tissues. (Growth variations in the activity of succinoxidase and succinidehydrazase of the liver and the brain.)" Khar'kov, 1960. 19 pp; (Ministry of Higher and Secondary Specialist Education Ukrainian SSR, Khar'kov Order of Labor Red Banner State Univ im A. M. Gor'kiy); 150 copies; free; (KL, 25-60, 129)

SAZONOVA, Ye.A.

Nature of age-connected changes in oxidation processes of tissues.
Report No.1: Changes with age in the succinic oxidase and succinic dehydrase activity of the liver. Uch. zap KGU 108:273-285 '60.

(MIRA 14:3)

1. Kafedra fiziologii cheloveka i zhivotnykh Khar'kovskogo gosudarstvennogo universiteta.

(OXIDATION, PHYSIOLOGICAL) (AGE) (LIVER)

SAZONOVА, Ye.A.

Nature of age-connected changes in oxidation processes of tissues.

Report No.2: Changes with age in the succinic oxidase and succinic dehydrase activity of the brain. Uch. zap KНGU 108:287-291 '60.

(MIRA 14:3)

1. Kafedra fiziologii cheloveka i zhivotnykh Khar'kovskogo gosudarstvennogo universiteta.

(OXIDATION, PHYSIOLOGICAL) (AGE) (BRAIN)

TARARYKOV, G.M.; SAZONOV, Ye.A.

Use of brewery wastes for the product on of fodder terramycin.
Ferm.i spirt.prom. 31 no.1:31-33 '65.

(MIRA 18:5)

1. Voronezhskiy tekhnologicheskiy institut.

SAZNOVA, Ye. D.

"Influence of Chloranhydrides of Acids on Diethyl-Hydroxylamine," Zhur. Obshch. Khim.,
14, No. 3, 1944. All-Union Scientific Research Chemico-Pharmaceutical Inst., im. S.
Ordzhonikidze, -1643-.

with A. Ya. Berlin

SAL'NIKOVA, Ye. D.

"On the Production of 6-Ketox-4-(8Diethylamino-a-Methyl-Butyl)-Aminoquinoline," Zhur. Obshch. Khim., 16, No. 11, 1946. First Synthetics Dept., All-Union Sci. Research Chemico-Pharmaceutical Inst. im. S. Ordzhonikidze, Moscow, -1945-.

SAZONOVA, Ye. D.

Apr 49

USSR/CHEMISTRY- CHLORAL
ANILIDES

"The Condensation of Acylanilides With Chloral," A. Ya. Berlin, M. N. Shchukiha, Ye. D. Sazonova, All-Union Sci [les]es Chemicophar Inst imeni S. Ord-Zhonikidze, 6 pp

"Zhur Obshch Khim" Vol XIX, No 4

Study of subject reaction in the presence of H_2SO_4 , established that acetanilide and phthalanil enter into the reaction, while succinanil, in the observed experiments, did not. Gives products of the reaction of acetanilide and the phthalanil with the chloral.

PA 65/49T22

SAZONOVA, Ye. D.

"Org- and Mehtoxyarino-substituted alpha, alpha-Diphenyl-beta, beta, beta-trichloroethane",
Zhur. Obshch. Khim. 19, No. 11, 1949., All-Union Sci-Res Chern-Pharm Inst im S. Ordzhon-
ikidze, Moscow. -c1949-.

10

Hydroxy- and methoxyamino-substituted 2,2-diphenyl-
1,1,1-trichloroethane. M. N. Sizukina and E. D.

Sazonova. (All-Union Research Inst. Pharm. Chem.,
Moscow). *J. Gen. Chem. U.S.S.R.* 19, No. 11, u505-70
(1949) (Engl. translation).—See *C.A.* **44**, 3952c.

E. J. C.

(1)

Hydroxy- and methoxyamino substituted derivatives of 2,2-diphenyl-1,1,1-trichloroethane. M. N. Shechukina and E. D. Sazonova (All Union Chem. Pharm. Research Inst., Moscow). *Zhur. obshch. Khim.* (J. Gen. Chem.). 19, 2004-9 (1949). — *p*-AcNH₂C₆H₄OMe (10.6 g.) stirred at -5° with 75 ml. 100% H₂SO₄ and 7.5 g. Cl₃CCHO, then 0.5 hr. at room temp., gave 13 g. [2,3-MeO(AcNH)-C₆H₄]CHCCl₂ (I), m. 220-7° (from EtOH), contg. BiOH or cryst.; boiling with dil. HCl 8 hrs. gave the free amino deriv., m. 74°; *di-HCl salt*, m. above 230° (from EtOH). — *o*-AcNH₂C₆H₄OMe gave [4,3-MeO(AcNH)-C₆H₄]CHCCl₂, m. 213-11° (from EtOH, contains 1 EtOH); *free amine*, m. 60-1°; *di-HCl salt*. Similarly 7.5 g. *p*-AcNH₂C₆H₄OH gave 10.2 g. [4,3-HO(AcNH)-C₆H₄]CHCCl₂, m. 220-7° (from 0.5% EtOH); *free amine*, solid (from C₆H₆), decomp. 152°. The *p*-isomer gave only *di-acetamido-2,2-bis(trichloromethyl)-1,2-benzodioxane*, m. 205° (cf. Chattaway, C.I. 21, 250). Boiling 2 g. 12 hrs. with 10 ml. AcOH and 1 g. 92% H₂SO₄ gave 1.6 g. poorly sol. *di-HCl salt* of the *di-HO* analog, m. 242°; *free amine*, m. 175° (from C₆H₆), is poorly stable. A reaction analogous to the prepn. of I with 7.5 g. *m*-AcNH₂C₆H₄OH gave 4 g. [4,2-HO(AcNH)-C₆H₄]CHCCl₂, m. 180° (from C₆H₆-EtOH), which could not be decarbonylated without decomp., but treatment with LiONa and Mel in EtOH gave the *di-MeO* analog, m. 129° (from dil. EtOH). Analogously, *m*-MeOC₆H₄NHAc gave 2,4-ArNH(MeO)-C₆H₄CH(OH)CCl₂, m. 179° (from EtOH); hydrolysis with 17% HCl gave the *HCl salt* of the *free amine*. — G. M. K.

SAZNOVA, Ye. D. and SHCHUKINA, M. N.

Synthesis of N-Substituted p-Aminobenzal Thiosemicarbazones, page 1081,
Sbornik statey po obshchey khimii (Collection of Papers on General Chemistry),
Vol II, Moscow-Leningrad, 1953, pages 1680-1686.

All-Union Sci Res Chemico-Pharmaceutical Instiimeni S. Ordzhonikidze

SAZONOVA, YE. D.

USSR/Chemistry - Pharmaceuticals

Sep 52

"Some Syntheses in the Series of 4-Acetaminobenzaldehydes," M. N. Shchukina, G. M. Borodina, Ye. D. Sazonova, All-Union Sci Res Chem-Phar Inst imeni S. Ordzhonikidze, Moscow

"Zhur Obshch Khim" Vol 22, No 9, pp 1659-1663

A series of aromatic aldehydes contg substituents in the 2 and 3 positions as well as an acetamino group in the 4 position were obtained. The thiosemicarbazones of these aldehydes were prep'd. By treating 4-amino-2- and 3-methoxybenzaldehydes with succinic anhydride or benzoyl chloride, the corresponding acylated compds were obtained. The prepn of 4-acetamino-2methoxycinnamic acid, 4-acetamino-2-methoxyhydroxycinnamic acid, and 4-amino-2-oxyhydrocinnamic acid is described.

232T31

Sazonova, E. D.

CATALYSTS

Chemical Abst.
Vol. 48 No. 8
Apr. 25, 1954
Organic Chemistry

Some syntheses in the α -acetamido- β -phenylaldehyde series.
M. N. Soshnikina, G. M. Berdina, and E. D. Sazonova,
J. Gen. Chem. (U.S.S.R.) 22, 1701-4 (1952) (Engl. transla-
tion) — See C.A. 47, 9298d

(3) fuel

H. L. H.

8-30-54
298

SAZONOVA, YE. D.

USSR/Chemistry - Pharmaceuticals
Medicine - Tuberculosis, Chemotherapy

11 Jun 52

"Isonicotinoyl Hydrazones and Their Antitubercular Activity," M. N. Shchukina,
G. N. Pershin, O. O. Makeyeva, Ye. D. Sazonova, Ye. S. Nikitskaya, A. D. Yanina,
A. I. Yakovleva, All-Union Sci Res Chem-Phar Inst imeni S. Ordzhonikidze

"Dok Ak Nauk SSSR" Vol LXXXIV, No 5, pp 981 - 984

Isonicotinoyl hydrazide has an antitubercular action, but its therapeutic index is low. A series of substituted isonicotinoyl hydrazide derivs were prep'd and their bacteriostatic action on tuberculosis bacilli in vitro tested. It was shown that substituted isonicotinoyl hydrazones have a higher antitubercular activity than para-aminosalicylic acid and streptomycin and are better tolerated by exptl animals than the hydrazide of isonicotinic acid. This made it possible to select from them substances for clinical study on tubercular humans. A substance, called "Phtivaccine" by the authors, was forwarded for clinical study, which is progressing successfully. Presented by Acad A.N. Nesmeyanov 9 Apr 52

223T17

Synthesis of *N*-substituted *p*-aminobenzalthiosemicarbazones. M. N. Shchukina and E. D. Sazonova (S. G. Orloshnikidze All-Union Chem. Pharm. Research Institute).

Sbornik Statei Obzhetel Khim., 2, 1031-4 (1953). — To refluxing soln. of 100 g. *p*-O₂NC₆H₄Me in 100 ml. EtOH was added in 1.5 hrs. a hot soln. of 60 g. I in 825 ml. 12% NaOH, the mixt. stirred and heated 3 hrs. longer, cooled to 30°, the upper layer sepd. and treated with a hot soln. of 42 g. thiourea in 250 ml. H₂O, heated 15 min., cooled to 20°, and neutralized with 40 ml. 80% AcOH, yielding 69.3% *p*-aminobenzalthiosemicarbazone (I), m. 107-8° (from 2% HCl). This treated with Ac₂O in Me₂CO 3-4 hrs. gave 92.1% *Ac deriv.*, m. 224-6°. To 1.4 g. ClCH₂CO₂H in 1.5 g. Na₂CO₃ and 30 ml. H₂O was added 1.04 g. I and the mixt. refluxed 2.5 hrs. and cooled, yielding 94% *p*-glycyl-aminobenzalthiosemicarbazone, m. 270-1° (from 50% AcOH); this (3.88 g.) in 100 ml. EtOH was treated with 3.8 g. glucose in 25 ml. H₂O and 0.15 g. 40% NaHSO₃ and refluxed 4 hrs.; on cooling there is obtained a ppt. of 40% colorless *glucose bisulfite deriv.*, CuH₁₁N₂O₄S₂Na, m. 221° (from EtOH). Heating 19.1 g. I with 15 g. formaldehyde NaHSO₃ complex and 0.5 ml. 40% NaHSO₃ (solvent not specified) 1 hr. gave 70.4% *I formaldehyde bisulfite complex*, CuH₁₁N₂O₄S₂Na·H₂O, decomp. 225° (from H₂O). Heating 3 g. I with 2.2 g. Et-NCH₂CH₂Cl 3 hrs. at 80° gave 3.2 g. *p*-2-diethylaminoethyl-aminobenzalthiosemicarbazone, yellow, m. 186-7° (from H₂O). Addn. of 1.1 g. (CH₃CO)₂O to 1.94 g. I in Me₂CO gave a ppt. of 85% *p*-succinylaminobenzalthiosemicarbazone, m. 199-200° (from EtOH). I with BzCl similarly gave the *p*-benzamido deriv., 83.9%, decomp. 267-8° (from EtOH). Nicotinoyl chloride in C₆H₅pyridine gave with p-

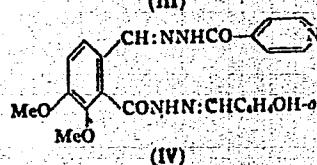
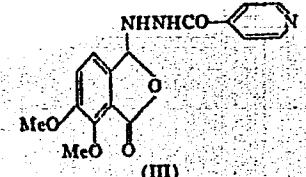
DOVER

$\text{H}_2\text{NC}_6\text{H}_4\text{CHO}$, colorless *p*-nitroanilinobenzaldehyde, m.
174-5° (from H_2O); *thiocarbazone*, decomp.
(from EtOH). Heating 5 g. *p*-aminobenzylic acid with 2
ml. Ac₂O 2 hrs. gave 78% 2-acetyl-1-acetamidobenzoic acid,
m. 189-90° (from EtOH). This (3 g.) heated 3 hrs. at 60°
with 5 g. SOCl₂, evapd. *in vacuo*, and the residual acyl
chloride treated with 1.5 g. *p*-H₂NC₆H₄CHO in C₆H₆ and 2
ml. pyridine; and refluxed 3 hrs. gave 0.5 g. 4-(2-hydroxy-4-
acetamidobenzoyl)anilinobenzaldehyde, m. 214-18° (from
 H_2O); *thiocarbazone*, decomp. 235-0° (from EtOH).

G. M. Koslapoff

Syntheses in the series of derivatives of *benzimidazole* and its hydrochloride. M. N. Stachukina and E. D. Surovaya (S. D. Shchegoleva). Phosphorus-Substituted *benzimidazole*. All-Union Chem.-Physic. Inst., Moscow. Zhur. Russ. Khim. Kharakteristika. 23, 637-644 (1958); c. A. 64, 10431B.

Opionic acid [$\text{C}_8\text{H}_7\text{O}_4\text{C}(\text{MeO})_2\text{C}_6\text{H}_4\text{CHO}$] (2.1 g.) in 40 ml. hot H_2O added a hot soln. of 1.37 g. ioniconic hydrazide (I) gave on cooling 98% ioniconic hydroxydrazone (II), m. 208-8° (*monohydrate*, loses H_2O at 160°); *Na salt*, crystals (from H_2O), m. 230-40°; diethanolamine salt, m. 186-7° (from EtOH); Et_2NH salt, m. 178-7° (from EtOH). Aq. solns. of these salts become cloudy on standing and deposit *ppgs.* of the salts. Addn. of 1.37 g. I in 10 ml. H_2O to 2.1 g. Et pseudopionate [$\text{C}_8\text{H}_7\text{O}_4\text{C}(\text{MeO})_2\text{C}_6\text{H}_4\text{CH}_2\text{CO}_2\text{Et}$] gave a cyclic compd. (IIIa), m. 207-8°. Heating II in EtOH gave III. IIa and III appear to be the same compd., but a mixed m.p. depresses to 190-8°. Similarly was prep'd.



2-carboxy-3,4-dimethoxybenzaldehyde *nicotinoylhydrazone* (IIb), (97%), m. 214-16° (monohydrate, loses H₂O at 150°). II (3.2 g.) heated 3 hrs. in 50 ml. abs. EtOH satd. with HCl gave 2.6 g. *Et* ester of II, m. 213-14° (from EtOH), which with N₂H₄·H₂O rapidly gave 87% 2-NH₂NHCO analog, m. 152-3° (from H₂O). This (1.5 g.) in EtOH with 0.6 g. o-HOC₂HCHO in 10 ml. hot EtOH gave 1.9 g. 4-(o-HO-C₂HCH₂NHCO) anolog (m. 210-11°) (from EtOH). Heating 10 g. 4-chloropyrazine [cf. Gabriel, *Ber.*, 36, 3378 (1903)] and 20 g. N₂H₄·H₂O to 30° led to a spontaneous reaction and on cooling the mist yielded 4-hydrazino-5,6-dimethoxyphthalazine, m. 75-8°; HCl salt, m. 164-6° (from EtOH). Heating 2.25 g. 4-chloropyrazine with 1.37 g. I in EtOH 1 hr. gave 90% 5,6-dimethoxy-4-(isocitroinoylhydrazino)phthalazine, decomposed 234-5° (from EtOH). Similarly was prep'd. the nicotinoylhydrazine isomer (90%), m. 207-8°. The latter and III are less active against the tuberculous bacteria than the corresponding isonicotinic derivs. Introduction of a 2nd -CNH₂C group into the isonicotinoylhydrazones reduces their activity. G. 14. K.

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✓Synthesis in the series of derivatives of isonicotinic acid hydrazide. M. N. Shchukina and B. D. Sazonova,
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Aromatic isonicotinylhydrazones; a new class of drugs in the treatment of tuberculosis. Probl.tub. no.2:44-50 Mr-ap '54.
(MLRA 7:5)

1. Iz Vsesoyuznogo nauchno-issledovatel'skogo khimiko-farmatsev-ticheskogo instituta.

(TUBERCULOSIS, experimental,

*eff. of isonicotinaldehyde thiosemicarbazone)

(ALDEHYDES, effects,

*isonicotinaldehyde thiosemicarbazone, on exper. tuberc.)

(THIOSEMICARBAZONES, effects,

*isonicotinaldehyde thiosemicarbazone, on exper. tuberc.)

SAZONOVA, Ye.M.

Oxygen therapy in so-called cor pulmonale. Trudy LSGMI
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1. Fakul'tetskaya terapevicheskaya klinika Leningradskogo
sanitarno-gigiyenicheskogo meditsinskogo instituta (zav.
klinikoy - prof.A.A.Kedrov).

(PULMONARY HEART DISEASE, ther.

oxygen (Rus))

(OXYGEN, ther. use,

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1. Department of Human and Animal Physiology of the A.M. Gorkiy State
University of Kharkov.
(SUCCINIC OXIDASE) (SUCCINIC DEHYDROGENASE)

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1. Nauchno-issledovatel'skiy institut udobreniy i insektofung-
isidov.